

# AFRL-RX-WP-TM-2008-4368

# THERMOCHEMICAL AND PHYSICAL PROPERTIES OF FLUIDS, LUBRICANTS AND RELATED MATERIALS

Delivery Order 0001: Improved Methods Development for Determining Thermochemical Properties of Fluids, Lubricants and Related Materials

Arthur A. Krawetz

**Phoenix Chemical Laboratory, Inc.** 

**JULY 2008** 

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#### 14. ABSTRACT

A new method is described that determines the corrosion protection of fluids, lubricants and related materials and automatic equipment for the measurement thereof.

Chemical and physical properties of experimental hydraulic fluids, dielectric coolants, grease and related materials are reported.

#### 15. SUBJECT TERMS

Liquid lubricants, grease, hydraulic fluids, coolants, lubricity, corrosion protection, oxidative stability

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#### **Preface**

Contract No. F33615-03-D-5049. The work was initiated under Project No. 4347. The effort was administered under the direction of the Air Force Research Laboratory, Materials and Manufacturing Directorate, Nonmetallic Materials Division, Thermal Sciences and Materials Branch (AFRL/RXBT), Wright-Patterson Air Force Base, Ohio, with Carl Ed Snyder, Jr., as Project Engineer.

This report covers work performed between 19Feb2003 and 15August2008. The draft report was submitted by the author in August 2008.

A number of subjects have been addressed as part of the current effort. The main part of this report covers the study of an improved corrosion rate evaluation procedure and customized equipment for its determination. Appendix II of this report covers the relevant chemical and physical properties of lubricants and hydraulic fluids. The author acknowledges the contributions of several individuals who contributed to the experimental phases of the work. These include in alphabetical order, the following: Pricha Klinsuttho and Theodore Tovrog. Special recognition is extended to Theodore Tovrog whose counsel, advice and assistance were of invaluable assistance in the initial phase of this research effort. He retired on April 25, 2003.

The author specially acknowledges the assistance of Carol Wilson and Susan Ryman in the preparation of this report.

# 1. Summary

In this report a final Improved Corrosion Rate Evaluation Procedure and Automatic CREP System for determining the corrosion protection capabilities of candidate fluids and lubricants are described under Presentation of Results. In Appendix II, Presentation of related chemical and physical properties of lubricants and hydraulic fluids are provided.

#### 2. Introduction

Modern aircraft and missile systems operate at temperatures and pressures which expose their hydraulic fluids and lubricants to ever increasing stress. Demands for increased performance under such conditions have become a requirement of critical proportions. For this reason unconventional alloys and metals together with fluids and lubricants capable of functioning in that high stress environment have been sought. The evaluation of the performance expectancy of these unorthodox systems has become a concomitant problem. Classical means for the evaluation of such properties have not always proved adequate, although they retain great utility – especially for their ability to provide a continuing data base for the comparison of common elements of functional ability.

Standard procedures for the evaluation of the efficiency of rust productive agents have invoked prolonged exposure to humidity and salt spray in standardized cabinets. Occasionally, accelerating agents such as sulfur dioxide or hydrobromic acid have been employed to increase the severity of exposure and/or to expand to the extent of product screening within a given time frame. In that context the Corrosion Rate Evaluation Procedure (CREP) was developed (Reference 1). While that procedure provides for the rapid evaluation of rust preventive agents and produces data which have, at least, relative significance. The reproducibility of the method is poorer than that required for a procedure which can be adapted as a test method suitable for use in product specifications and requires a subjective assessment of the level of corrosion on the test specimens.

# 3. Methods and Procedures

In addition to the detailed description of the method and apparatus developed for corrosion rate evaluation of lubricants and hydraulic fluids data relevant to their use under extremes of physical, chemical and environmental stress are included.

#### 4. Presentation of Results and Discussion

# **4.1.** Improved Corrosion Rate Evaluation Procedure and Automatic CREP System for Corrosion Rate Evaluation

Modifications of the original CREP procedure have been previously reported (References 1, 2, and 3). It was the conclusion of those studies that measurements of weight loss of specimens exposed either to distilled water or buffer solution vapors produced results which were difficult to reproduce even under the most carefully controlled conditions. The visual rating of exposed specimens, although subjective, was found to present similar problems in addition to those naturally associated with any subjective method. "Good" and "bad" performance previously could only be determined by comparison of unknown performance with that of known specimens exposed simultaneously in the same cell as the unknown.

The procedure which is described in the following text, together with accompanying data which describe the final version of the test apparatus, present a repeatable means for the evaluation of corrosion rate which does not require the simultaneous use of standard replicates.

### 4.2 Operational Procedure for CREP-I

- 1. Place 20 mL of water or buffer solution as may be required in each test cell. Add one boiling stone to prevent uneven evaporation of test solution.
- 2. Turn on heater control and allow each cell to equilibrate for 60 minutes at 92±1°C. See Procedure for setting temperature (Appendix I). Note: liquid must be present in each cell.
- 3. Polish each metal test coupon as described in ASTM D1748, finishing with 320 grit aluminum oxide abrasive. Polish with even strokes being careful not to burnish the surface of the test specimens by the use of excessive pressure.
- 4.Rinse the test specimens with n-heptane or iso-octane to remove any loose particulate matter.
- 5. Wash each test specimen in boiling toluene for 5 minutes and in boiling acetone for 5 minutes.
- 6. Allow cleaned metal specimens to dry for 30 minutes at room temperature in a dust-free cabinet.
- 7. With a clean stainless steel hook dip each metal specimen in the oil to be tested and soak therein for 5 minutes.

- 8. Allow coated specimens to hang in a dust-free cabinet for 15 minutes to permit excess oil to drip free.
- 9. Transfer each coated specimen to the glass hook on the adapter fixture and gently touch the bottom of each specimen with an absorbent filter paper to remove any excess oil drops which may remain.
- 10. Assemble the test apparatus by placing the adapter on top of the test cell followed by the condenser placed on top of the adapter.
- 11. Adjust the potentiometer for each cell as follows:
- a. Turn each clockwise, if the running signal (green) is seen, and until the failing signal (orange) is just on; then turn each counter clockwise until the running signal is on; finally turn each counter clockwise 30 additional divisions.
- b.Turn each counter clockwise, if the failing signal (orange) is seen and until the running signal (green) is just on; finally turn each counter clockwise 30 additional divisions.
- 12. At the end of the test each, the orange failing signal will appear for 20 seconds after which the ending red light signal will turn on.
- 13. Optimal confirmation of the validity of the test result may be had anytime after the red light: press each toggle switch. Then, should the running signal reappear, the end point of the test is in doubt. If a false ending signal is, thus, indicated, repeat the test.
- 14.Remove coupons, wash with mineral spirits, and with acetone. Rate the appearance of the panel and record the final running time.

# **5. Conclusions**

The data which have been obtained provide a basis for the selection of newly developed lubricants and hydraulic fluids including those formulated with newly developed matrices which are capable in meeting the stresses encountered in high performance systems in an environmental sound manner.

#### 6. References

#### AFWL-TR-92-4020

A. A. Krawetz, Technical Report AFWL-TR-92-4020, Volume II, Thermochemical and Physical Behavior of Lubricants and Hydraulic Fluids, Air Force Systems Command (ASD), Wright Laboratory (ML/MLBT), Wright-Patterson Air Force Base, Ohio, May, 1992.

#### WL-TR-96-4090

A.A. Krawetz, Technical Report WL-TR-96-4090, Thermochemical and Physical Properties of Fluids, Lubricants and Related Materials, Volume 3, Improved Corrosion Rate Evaluation Procedure; Chemical and Physical Properties of Lubricants and Hydraulic Fluids, May, 1996.

#### **AFRL-ML-WP-TR-2002-4185**

A. A. Krawetz, Technical Report AFRL-ML-WP-TR-2002-4185, Thermochemical and Physical Properties of Fluids, Lubricants and Related Materials, Volume 1, Improved Corrosion Rate Evaluation Procedure, August, 2002.

# **List of Acronyms**

CREP Corrosion Rate Evaluation Procedure

CREP-I Improved Corrosion Rate Evaluation Procedure

CREP-A Automatic Corrosion Rate Evaluation Procedure

# Appendix I

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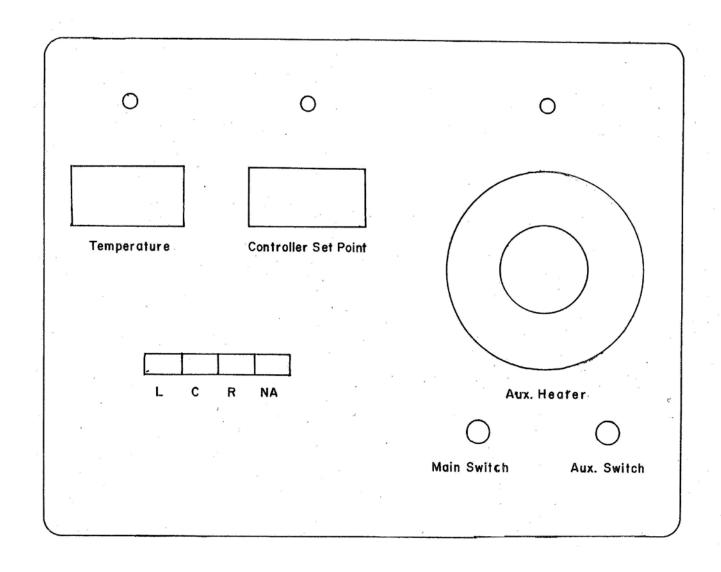


Figure 1 Heater Control Panel

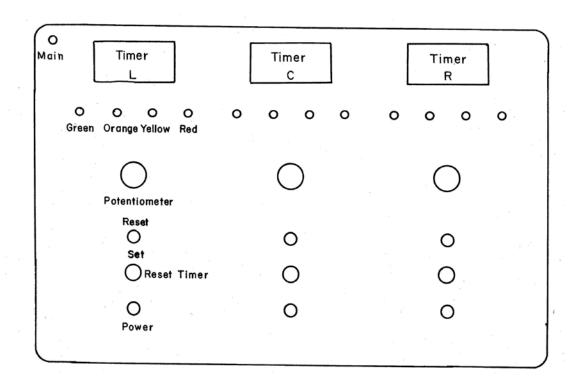


Figure 2 Sensor Control Panel

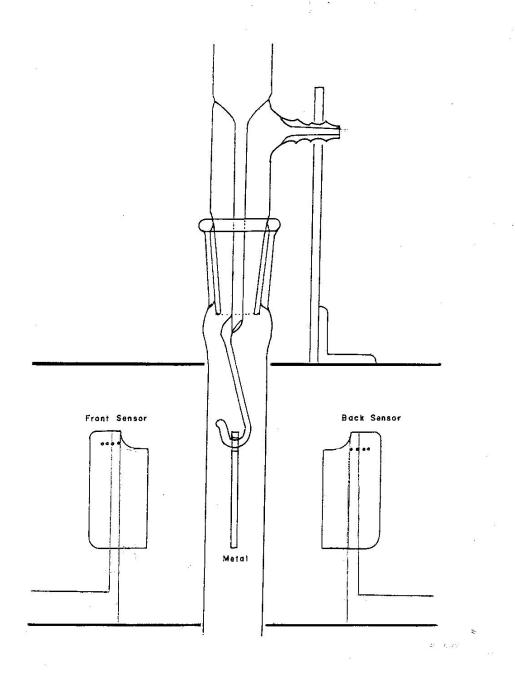


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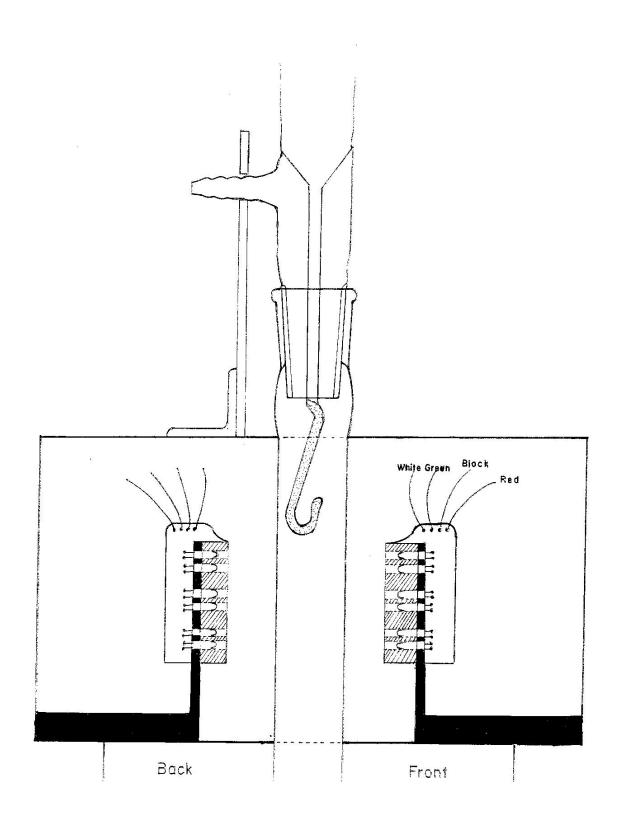


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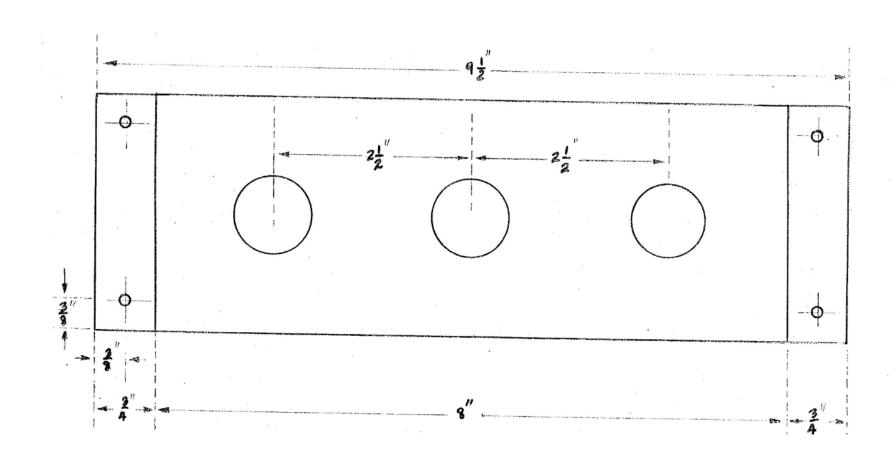


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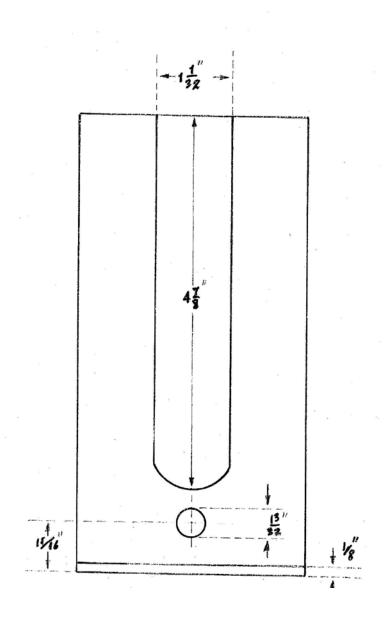


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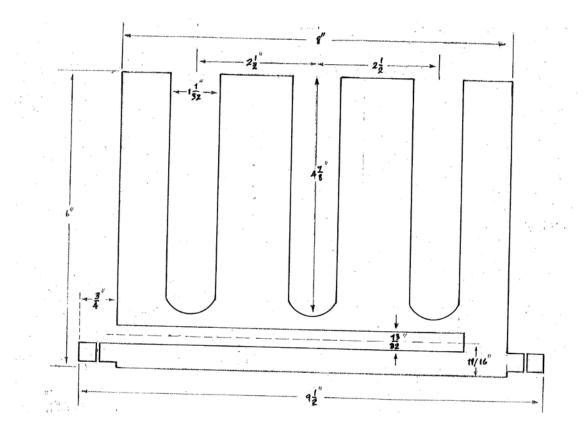


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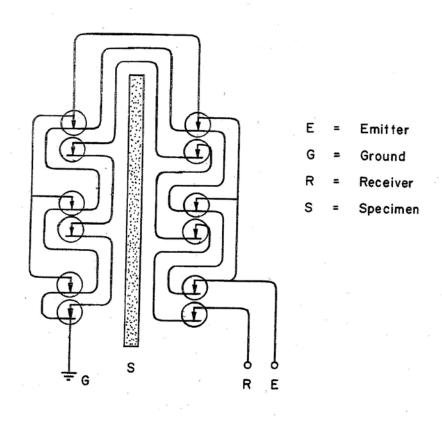
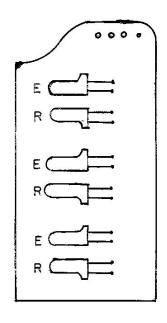


Figure 8 Schematic of Sensor Unit



- E iR-emitter(VTE3372LAH)
  3 mm Diameter, 20 mA
- R Phototransister (VTT 3323LAH) 3 mm Diameter, 25 mA

- E Emitter
- R Receiver

Figure 9 Schematic of Emitter- Receiver Panel

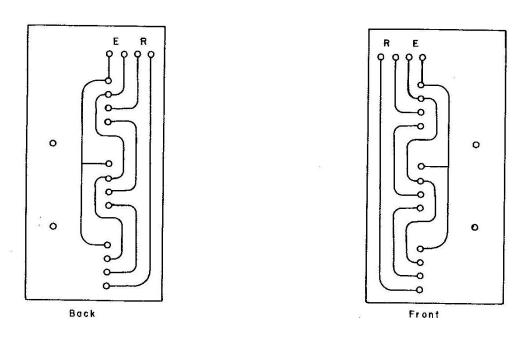


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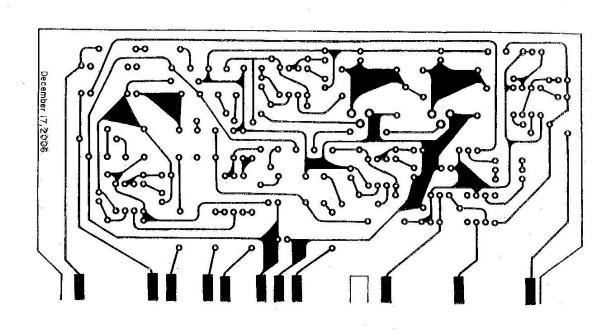


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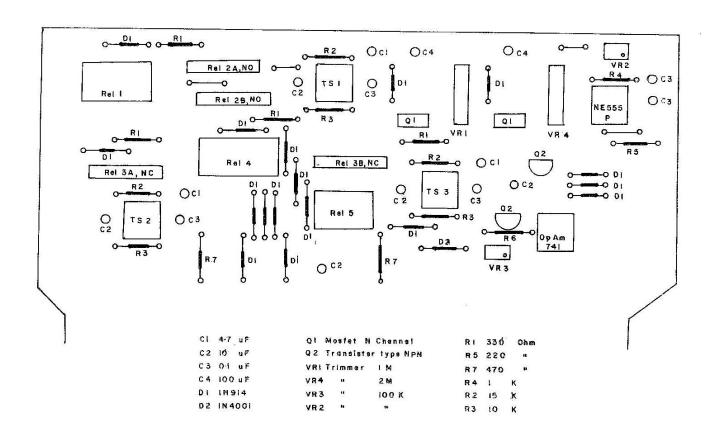


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### **Appendix II**

# THERMOCHEMICAL AND PHYSICAL PROPERTIES OF FLUIDS, LUBRICANTS AND RELATED MATERIALS

### CHEMICAL AND PHYSICAL PROPERTIES OF LUBRICANTS AND HYDRAULIC FLUIDS

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#### **Foreword**

This report was prepared by Phoenix Chemical Laboratory, Inc., under USAF Contract No. F33615-03-D-5049-0001. The work was administered under Project No. FAR52.204.03. The work was administered under the direction of the Material Directorate (AFRL/RXBT), Wright-Patterson Air Force Base, Ohio, with Carl E. Snyder, Jr., as Project Engineer.

This report covers work performed between 19 Feb.2003 and 15 August 2008. The report was submitted by the authors in September 2008.

A number of subjects have been addressed as part of the current effort: Part 2 of this report covers the measurement of the chemical and physical properties of a variety of candidate high performance materials. The author acknowledges the significant contributions of Pricha Klinsuttho, Gregory Musil, Isora Rodriguez, Vicente Rodriguez, Ludmilla Lutsky, and Boris Guberman to the accomplishment of the objectives of this research effort.

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**1.** Low Temperature Torque Determinations

TABLE 1
SAMPLE NUMBER MLO-94-23

# Low Temperature Torque of Ball Bearing Grease ASTM D1478, @-54°C(-65°F), Torque, gm

	Run 1	Run 2	Run 3	Average
Starting Torque, Maximum	17,552	16,018	16,815	16,795
Running Torque, 60 seconds	13,865	13,128	13,570	13,521
Running Torque, 10 minutes	9,588	10,178	9,971	9,912
Running Torque, 60 minutes	5,104	4,248	4,572	4,641

#### **SAMPLE NUMBER MLO 94-23**

Low '	Temperature	e Torqı	ıe
(AS	TM D1478)	@ -54	°C(-65°F)

Starting Torque, N.m	24.5
Running Torque (60 min), N.m	10.3

TABLE 3
SAMPLE NUMBER MLO-01-127

# Low Temperature Torque of Ball Bearing Grease ASTM D1478, @-54°C(-65°F), Torque, gm

	Run 1	Run 2	Run 3	Average
Starting Torque, Maximum	8,850	8,762	9,145	8,919
Running Torque, 60 seconds	6,578	6,549	6,342	6,490
Running Torque, 10 minutes	5,900	4,572	5,988	5,486
Running Torque, 60 minutes	2,272	2,242	2,360	2,291

TABLE 4
SAMPLE NUMBER MLO-01-232

# Low Temperature Torque of Ball Bearing Grease ASTM D1478, @-54°C(-65°F), Torque, gm

	Run 1	Run 2	Run 3	Average
Starting Torque, Maximum	4,868	4,278	4,425	4,524
Running Torque, 60 seconds	2,744	2,390	2,242	2,458
Running Torque, 10 minutes	738	944	1,092	924
Running Torque, 60 minutes	708	590	560	619

#### **SAMPLE NUMBER MLO 01-232**

Low Temperature Torque	
(ASTM D4693) @ -54°C(-65°F)	
Torque, N.m (60 sec):	1.6
Torque, max: (Start)	2.0

TABLE 6
SAMPLE NUMBER MLO-03-21

# Low Temperature Torque of Ball Bearing Grease ASTM D1478, @-54°C(-65°F), Torque, gm

	Run 1	Run 2	Run 3	Average
Starting Torque, Maximum	10,915	11,004	11,652	11,190
Running Torque, 60 seconds	8,702	8,054	8,555	8,437
Running Torque, 10 minutes	6,992	6,726	7,228	6,982
Running Torque, 60 minutes	2,478	2,655	2,655	2,596

#### **SAMPLE NUMBER MLO 03-48**

12,331

# Low Temperature Torque (ASTM D1478) @ -54°C(-65°F) Starting Torque, g-cm >14,750

Running Torque (60 min), g-cm

#### Corrected Report, discard previous report.

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Low Temperature Torque (ASTM D4693) @ -54°C(-65°F) Torque, N.m (60 sec): Torque, max: (Start)	7.4 15.2
SAMPLE NUMBER MLO 01-127	
Low Temperature Torque	
(ASTM D4693) @ -54°C(-65°F)	
Torque, N.m (60 sec):	5.6
Torque, max: (Start)	10.8
SAMPLE NUMBER MLO 94-23	
Low Temperature Torque	
(ASTM D4693) @ -54°C(-65°F)	
Torque, N.m (60 sec):	10.3
Torque, max: (Start)	24.5
1 / /	

#### **SAMPLE NUMBER MLO 03-48**

# Low Temperature Torque (ASTM D1478) @ -40°C (25# Dynomometer)

Starting Torque, g-cm	2360
Running Torque (1 min), g-cm	1534
Running Torque (10 min), g-cm	1210
Running Torque (60 min), g-cm	384

**TABLE 10** 

	MLO 94-23	MLO 01-127
Low Temperature Torque (ASTM D1478) @ -54°C (25 lb Dynamometer)		
Starting Torque, g-cm Running Torque (1 min), g-cm Running Torque (10 min), g-cm Running Torque (60 min), g-cm	18,142 12,242 10,768 10,178	10,325 8,555 5,900 2,802
	MLO 01-232	MLO 03-48
Low Temperature Torque (ASTM D1478) @ -54°C (25 lb Dynamometer)		
Starting Torque, g-cm Running Torque (1 min), g-cm Running Torque (10 min), g-cm Running Torque (60 min), g-cm	4,278 2,508 885 590	14,160 9,588 6,195 3,688

#### **Comment:**

It was observed that bearing temperatures differed from test chamber temperatures. An additional baffle was installed to prevent air flow from the cold sink from impinging directly on the test bearing. In addition, a thermocouple was installed to monitor actual grease temperature in the test bearing.

All tests conducted with the 5 lbf dynomometer gauge were conducted before these modifications were made. All subsequent determinations with the 25 lbf dynomometer gauge were conducted after modification of the test cell.

#### **SAMPLE NUMBER MLO 03-21**

Low Temperature Torque (ASTM D1478) @ -54°C(-65°F)

Starting Torque, g-cm	>14,750
Running Torque (10 min), g-cm	13,865
Running Torque (60 min), g-cm	11,358

TABLE 12

Low Temperature Torque of Ball Bearing Grease ASTM D1478, @-40°C(-40°F), Torque, g·m

Torque	<b>Starting Torque</b>	<b>Running Torque</b>	<b>Running Torque</b>	Running
minutes)	(Maximum)	(60 seconds)	(10 minutes)	(60
MLO-05-0606	>73,750	-	(Frozen solid)	-
MLO-05-0607	>73,500	-	(Frozen solid)	-
MLO-05-0608	>73,750	-	(Frozen solid)	-
MLO-05-0609	>73,750	-	(Frozen solid)	-
MLO-05-0610	1,475	1,121	885	796
MLO-05-0611	1,770	1,032	856	590

TABLE 13

Low Temperature Torque of Ball Bearing Grease ASTM D1478, @-54°C(-65°F), Torque, g'm

Torque	<b>Starting Torque</b>	<b>Running Torque</b>	<b>Running Torque</b>	Running
minutes)	(Maximum)	(60 seconds)	(10 minutes)	(60
MLO-05-0606	>73,750	-	(Frozen solid)	-
MLO-05-0607	>73,500	-	(Frozen solid)	-
MLO-05-0608	>73,750	-	(Frozen solid)	-
MLO-05-0609	>73,750	-	(Frozen solid)	-
MLO-05-0610	3,982	2,655	2,301	1,888
MLO-05-0611	22,125	7,375	2,655	1,268

# TABLE 14 SAMPLE NUMBER MLO 06-733

#### Low Temperature Torque, (ASTM D1478), MIL-PRF-32014 Torque, g.cm

Torque, g.em	@ -54°C
Starting Torque, g-cm	3,687
Running Torque (1 min), g-cm	2,065
Running Torque (10 min), g-cm	1,740
Running Torque (60 min), g-cm	502

#### **SAMPLE NUMBER MLO 06-734**

#### Low Temperature Torque, (ASTM D1478), MIL-PRF-32014 Torque, g.cm

Torque, g.em	@ -54°C
Starting Torque, g-cm	16,225
Running Torque (1 min), g-cm	16,225
Running Torque (10 min), g-cm	5,015
Running Torque (60 min), g-cm	4,425

2	Water	Washo	ut Deter	minations
<i>Z</i> .	water	wasno	ut Deter	minations

**TABLE 16** 

SAMPLE NUMBER	Water Washout (ASTM @ 38°C @	* *
MLO-05-0606	20.2	42.4
MLO-05-0607	4.6	27.0
MLO-05-0608	18.1	37.0
MLO-05-0609	23.9	46.8
MLO-05-0610	0.00	0.20
MLO-05-0611	0.86	0.92

	MLO 06-733	MLO 06-734
Water Washout		
(ASTM D1264)		
@ 41°C, %	2.32	2.14

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#### **SAMPLE NUMBER MLO 03-25**

# Sonic Shear MIL-PRF-5606

#### **Properties of Reference Fluid A**

Viscosity @ 40°C, cs	
Before irradiation	57.82
After irradiation of 30 mL of fluid for 5 min.	50.85
Change, %	12.1
<b>Properties of Lab No. MLO 03-25</b> Viscosity @ 40°C, cs	
Before irradiation	35.06
After irradiation of 30 mL of fluid for 30 min.	34.66
Change, %	1.1

#### **SAMPLE NUMBER MLO 03-26**

## Sonic Shear MIL-PRF-5606H

#### **Properties of Reference Fluid B** Viscosity @ 40°C, cs Before irradiation 14.79 After irradiation of 30 mL of fluid for 30 min. 12.56 Change, % 15.1 Properties of Lab No. MLO 03-26 Viscosity @ 40°C, cs Before irradiation 14.21 After irradiation of 30 mL of fluid for 30 min. 12.31 Change, % 13.4

#### **SAMPLE NUMBER MLO 03-26**

#### Sonic Shear – Run 1 MIL-PRF-5606H (ASTM D5621)

#### **Properties of Reference Fluid B**

Viscosity @ 40°C, cs	
Before irradiation	14.06
After irradiation of 30 mL of fluid for 12.5 min.	11.96
Change, %	14.9
Properties of Lab No. MLO 03-26 Viscosity @ 40°C, cs	
Before irradiation	14.12
After irradiation of 30 mL of fluid for 30 min.	11.42
Change, %	19.1

#### **SAMPLE NUMBER MLO 03-26**

#### Sonic Shear – Run 2 MIL-PRF-5606H (ASTM D5621)

# Properties of Reference Fluid B Viscosity @ 40°C, cs Before irradiation 14.79 After irradiation of 30 mL of fluid for 12.5 min. 12.52 Change, % 14.3 Properties of Lab No. MLO 03-26 Viscosity @ 40°C, cs Before irradiation 14.21 After irradiation of 30 mL of fluid for 30 min. 11.08

22.0

Change, %

#### **SAMPLE NUMBER MLO 03-27**

## Sonic Shear MIL-PRF-5606H

#### Properties of Reference Fluid B Viscosity @ 40°C, cs Before irradiation 14.79 After irradiation of 30 mL of fluid for 30 min. 12.56 Change, % 15.1 Properties of Lab No. MLO 03-27 Viscosity @ 40°C, cs Before irradiation 14.22 After irradiation of 30 mL of fluid for 30 min. 12.49 Change, % 12.2

#### **SAMPLE NUMBER MLO 03-27**

#### Sonic Shear – Run 1 MIL-PRF-5606H (ASTM D5621)

#### Proportion of Deference Fluid R

Viscosity @ 40°C, cs	
Before irradiation	14.06
After irradiation of 30 mL of fluid for 12.5 min.	11.96
Change, %	14.9
Properties of Lab No. MLO 03-27 Viscosity @ 40°C, cs	
Before irradiation	14.31
After irradiation of 30 mL of fluid for 30 min.	11.47
Change, %	19.8

#### **SAMPLE NUMBER MLO 03-27**

#### Sonic Shear – Run 2 MIL-PRF-5606H (ASTM D5621)

#### **Properties of Reference Fluid B** Viscosity @ 40°C, cs Before irradiation 14.79 12.52 After irradiation of 30 mL of fluid for 12.5 min. Change, % 15.3 Properties of Lab No. MLO 03-27 Viscosity @ 40°C, cs Before irradiation 14.22 After irradiation of 30 mL of fluid for 30 min. 11.05 Change, % 22.3

#### **SAMPLE NUMBER MLO 04-1210**

# Sonic Shear MIL-PRF-5606

#### Properties of Reference Fluid B (MLO 82-208) Viscosity @ 40°C, cs Before irradiation 14.79 After irradiation of 30 mL of fluid for 30 min. 12.52 Change, % 15.3 Properties of Lab No. MLO 04-1210 Viscosity @ 40°C, cs Before irradiation 14.09 After irradiation of 30 mL of fluid for 30 min. 12.34 Change, % 2.4

4. Rubber Swell Test Determinat
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#### **SAMPLE NUMBER MLO-03-25**

#### Rubber Swell Test (ASTM D471) 70 Hours @ 205°C using 9218-410 O-Rings

#### **SWELLING**

Relative Density		% Swelling
1.500	1 <sup>st</sup> Specimen	3.39
1.502	2 <sup>nd</sup> Specimen	4.03
1.502	3 <sup>rd</sup> Specimen	3.90
1.501	Average	3.78

#### **ELONGATION**

% Elongation BEFORE Test		% Elongation AFTER Test
50	1 <sup>st</sup> Specimen	50
50	2 <sup>nd</sup> Specimen	50
50	3 <sup>rd</sup> Specimen	38
50	Average	46
	Change, %	-8.0

Tensile Strength BEFORE Test,	Tensile Strength AFTER Test,	
psi:	psi:	
833	1 <sup>st</sup> Specimen	1000
833	2 <sup>nd</sup> Specimen	1000
833	3 <sup>rd</sup> Specimen	916
833	Average	972
	Change, %	+16.7

#### **SAMPLE NUMBER MLO-03-25**

#### Rubber Swell Test (ASTM D471) 70 Hours @ 205°C using 9125-731 O-Rings

#### **SWELLING**

Relative Density		% Swelling
1.822	1 <sup>st</sup> Specimen	17.19
1.825	2 <sup>nd</sup> Specimen	17.40
1.825	3 <sup>rd</sup> Specimen	17.28
1.824	Average	17.29

#### **ELONGATION**

% Elongation BEFORE Test		% Elongation AFTER Test
200	1 <sup>st</sup> Specimen	62
200	2 <sup>nd</sup> Specimen	50
200	3 <sup>rd</sup> Specimen	50
200	Average	54
	Change, %	-73.0

Tensile	Strength AFTER Test,
	psi:
1 <sup>st</sup> Specimen	1912
2 <sup>nd</sup> Specimen	1912
3 <sup>rd</sup> Specimen	1764
Average	1862
Change, %	-13.1
	1 <sup>st</sup> Specimen 2 <sup>nd</sup> Specimen 3 <sup>rd</sup> Specimen Average

#### **SAMPLE NUMBER MLO-03-25**

#### Rubber Swell Test (ASTM D471) 70 Hours @ 205°C using 9125-777 O-Rings

#### **SWELLING**

Relative Density		% Swelling
1.810	1 <sup>st</sup> Specimen	12.73
1.810	2 <sup>nd</sup> Specimen	12.07
1.810	3 <sup>rd</sup> Specimen	11.87
1.810	Average	12.22

#### **ELONGATION**

% Elongation BEFORE Test		% Elongation AFTER Test
150	1 <sup>st</sup> Specimen	75
150	2 <sup>nd</sup> Specimen	125
150	3 <sup>rd</sup> Specimen	75
150	Average	92
	Change, %	-38.7

Tensile Strength BEFORE Test,	Tensile	Strength AFTER Test,
psi:		psi:
2500	1 <sup>st</sup> Specimen	2647
2500	2 <sup>nd</sup> Specimen	3970
2500	3 <sup>rd</sup> Specimen	2058
2500	Average	2892
	Change, %	+15.7

#### **SAMPLE NUMBER MLO-03-25**

#### Rubber Swell Test (ASTM D471) 70 Hours @ 70°C (158°F) using 9218-410 rubber specimens

#### **SWELLING**

Relative Density		% Swelling
1.504	1 <sup>st</sup> Specimen	5.46
1.502	2 <sup>nd</sup> Specimen	5.41
1.501	3 <sup>rd</sup> Specimen	5.39
1.502	Average	5.42

#### **ELONGATION**

% Elongation BEFORE Test		% Elongation AFTER Test
50	1 <sup>st</sup> Specimen	50
50	2 <sup>nd</sup> Specimen	75
50	3 <sup>rd</sup> Specimen	40
50	Average	55
	Change, %	10.0

Tensile Strength BEFORE Test,	Tensile Strength AFTER Test,	
psi:	psi:	
833	1 <sup>st</sup> Specimen	1166
833	2 <sup>nd</sup> Specimen	2000
833	3 <sup>rd</sup> Specimen	833
833	Average	1333
	Change, %	60.0

#### **SAMPLE NUMBER MLO-03-25**

#### Rubber Swell Test (ASTM D471) 70 Hours @ 70°C (158°F) using 9125-731 rubber specimens

#### **SWELLING**

Relative Density		% Swelling
1.827	1 <sup>st</sup> Specimen	4.90
1.826	2 <sup>nd</sup> Specimen	4.74
1.825	3 <sup>rd</sup> Specimen	4.98
1.826	Average	4.87

#### **ELONGATION**

% Elongation BEFORE Test		% Elongation AFTER Test
200	1 <sup>st</sup> Specimen	90
200	2 <sup>nd</sup> Specimen	90
200	3 <sup>rd</sup> Specimen	150
200	Average	110
	Change, %	-45.0

Tensile	Strength AFTER Test,
	psi:
1 <sup>st</sup> Specimen	5294
2 <sup>nd</sup> Specimen	5147
3 <sup>rd</sup> Specimen	4118
Average	4853
Change, %	126.6
	1 <sup>st</sup> Specimen 2 <sup>nd</sup> Specimen 3 <sup>rd</sup> Specimen Average

#### **SAMPLE NUMBER MLO-03-25**

#### Rubber Swell Test (ASTM D471) 70 Hours @ 70°C (158°F) using 9125-777 rubber specimens

#### **SWELLING**

Relative Density		% Swelling
1.812	1 <sup>st</sup> Specimen	5.57
1.816	2 <sup>nd</sup> Specimen	5.40
1.812	3 <sup>rd</sup> Specimen	5.47
1.814	Average	5.48

#### **ELONGATION**

% Elongation BEFORE Test		% Elongation AFTER Test
150	1 <sup>st</sup> Specimen	200
150	2 <sup>nd</sup> Specimen	75
150	3 <sup>rd</sup> Specimen	150
150	Average	142
	Change, %	-5.6

Te	ensile Strength AFTER Test,
	psi:
1 <sup>st</sup> Specimen	4705
2 <sup>nd</sup> Specimen	2647
3 <sup>rd</sup> Specimen	4412
Average	3921
Change, %	56.9
	1 <sup>st</sup> Specimen 2 <sup>nd</sup> Specimen 3 <sup>rd</sup> Specimen Average

#### **SAMPLE NUMBER MLO-03-275**

#### Rubber Swell Test (ASTM D471) 70 Hours @ 205°C using 9218-410 Rubber Specimens

#### **SWELLING**

Relative Density		% Swelling
1.498	1 <sup>st</sup> Specimen	6.20
1.498	2 <sup>nd</sup> Specimen	5.95
1.499	3 <sup>rd</sup> Specimen	6.03
1.498	Average	6.06

#### **ELONGATION**

% Elongation BEFORE Test		% Elongation AFTER Test *
50	1 <sup>st</sup> Specimen	50
50	2 <sup>nd</sup> Specimen	25
50	3 <sup>rd</sup> Specimen	75
50	Average	50
	Change, %	0.0

Tensile Strength BEFORE Test,	Tensile S	Strength AFTER Test,
psi:		psi: *
833	1 <sup>st</sup> Specimen	500
833	2 <sup>nd</sup> Specimen	416
833	3 <sup>rd</sup> Specimen	416
833	Average	444
	Change, %	-46.7

<sup>\*</sup> Specimen broke immediately at the jaws.

#### **SAMPLE NUMBER MLO-03-275**

## Rubber Swell Test (ASTM D471) 70 Hours @ 205°C using 9125-731 Rubber Specimen

#### **SWELLING**

Relative Density		% Swelling
1.815	1 <sup>st</sup> Specimen	34.66
1.819	2 <sup>nd</sup> Specimen	34.80
1.819	3 <sup>rd</sup> Specimen	34.54
1.818	Average	34.67

#### **ELONGATION**

% Elongation BEFORE Test		% Elongation AFTER Test *
200	1 <sup>st</sup> Specimen	50
200	2 <sup>nd</sup> Specimen	50
200	3 <sup>rd</sup> Specimen	50
200	Average	50
	Change, %	-75.0

#### TENSILE STRENGTH

Tensile Strength BEFORE Test,	Tensile	e Strength AFTER Test,
psi:		psi: *
2142	1 <sup>st</sup> Specimen	714
2142	2 <sup>nd</sup> Specimen	571
2142	3 <sup>rd</sup> Specimen	571
2142	Average	619
	Change, %	-71.1

<sup>\*</sup> Specimen broke immediately at the jaws.

#### **SAMPLE NUMBER MLO-03-275**

## Rubber Swell Test (ASTM D471) 70 Hours @ 205°C using 9125-777 Rubber Specimen

#### **SWELLING**

Relative Density		% Swelling
1.805	1 <sup>st</sup> Specimen	17.01
1.806	2 <sup>nd</sup> Specimen	17.10
1.806	3 <sup>rd</sup> Specimen	17.23
1.806	Average	17.11

#### **ELONGATION**

% Elongation BEFORE Test		% Elongation AFTER Test
150	1 <sup>st</sup> Specimen	150
150	2 <sup>nd</sup> Specimen	125
150	3 <sup>rd</sup> Specimen	150
150	Average	142
	Change, %	-5.6

#### TENSILE STRENGTH

Tensile Strength BEFORE Test,	Tensile	Strength AFTER Test,
psi:		psi:
2500	1 <sup>st</sup> Specimen	2142
2500	2 <sup>nd</sup> Specimen	2000
2500	3 <sup>rd</sup> Specimen	2000
2500	Average	2047
	Change, %	-18.1

# TABLE 35 SAMPLE NUMBER MLO-04-279

# Rubber Swell Test (ASTM D471) 70 Hours @ 70°C using 9218-410 O-rings

#### **SWELLING**

	<b>Relative Density</b>	% Swelling
Specimen #1	1.500	5.12
Specimen #2	1.501	5.22
Specimen#3	1.502	5.25
Average:	1.501	5.20

	BEFORE TEST	AFTER TEST
Specimen #1	98.6	98.3
Specimen #2	98.5	98.8
Specimen#3	98.3	98.0
Average:	98.5	98.4
Change:		01

TABLE 36
SAMPLE NUMBER MLO-04-279

# Rubber Swell Test (ASTM D471) 70 Hours @ 70°C using 9125-731 O-rings

#### **SWELLING**

	<b>Relative Density</b>	% Swelling
Specimen #1	1.820	4.63
Specimen #2	1.822	4.62
Specimen#3	1.825	4.43
Average:	1.819	4.56

	BEFORE TEST	AFTER TEST
Specimen #1	100.0	98.7
Specimen #2	100.0	100.0
Specimen#3	99.6	100.0
Average:	99.9	99.6
Change:		-0.3

**TABLE 37** 

#### **SAMPLE NUMBER MLO-04-279**

# Rubber Swell Test (ASTM D471) 70 Hours @ 70°C using 9125-777 O-rings

#### **SWELLING**

	<b>Relative Density</b>	% Swelling
Specimen #1	1.810	5.65
Specimen #2	1.812	5.38
Specimen#3	1.812	5.33
Average:	1.811	5.45

	BEFORE TEST	AFTER TEST
Specimen #1	100.0	99.8
Specimen #2	100.0	100.0
Specimen#3	100.0	99.7
Average:	100.0	99.8
Change:		-0.2

# TABLE 38 SAMPLE NUMBER MLO-04-279

# Rubber Swell Test (ASTM D471) 70 Hours @ 205°C using 9218-410 O-rings

#### **SWELLING**

	<b>Relative Density</b>	% Swelling
Specimen #1	1.500	5.11
Specimen #2	1.500	5.02
Specimen#3	1.503	5.21
Average:	1.501	5.11

	BEFORE TEST	AFTER TEST
Specimen #1	99.0	92.3
Specimen #2	99.2	93.0
Specimen#3	99.3	93.2
Average:	99.2	92.8
Change:		-6.3

TABLE 39
SAMPLE NUMBER MLO-04-279

# Rubber Swell Test (ASTM D471) 70 Hours @ 205°C using 9125-731 O-rings

#### **SWELLING**

	<b>Relative Density</b>	% Swelling
Specimen #1	1.820	32.64
Specimen #2	1.819	32.73
Specimen#3	1.819	32.67
Average:	1.819	32.68

	BEFORE TEST	AFTER TEST
Specimen #1	98.5	98.4
Specimen #2	99.1	99.1
Specimen#3	98.2	99.0
Average:	98.6	98.8
Change:		0.2

#### **SAMPLE NUMBER MLO-04-279**

# Rubber Swell Test (ASTM D471) 70 Hours @ 205°C using 9125-777 O-rings

#### **SWELLING**

	<b>Relative Density</b>	% Swelling
Specimen #1	1.822	16.32
Specimen #2	1.819	16.62
Specimen#3	1.824	16.60
Average:	1.822	16.52

	BEFORE TEST	AFTER TEST
Specimen #1	99.2	99.3
Specimen #2	100.0	99.4
Specimen#3	99.3	99.2
Average:	99.5	99.3
Change:		-0.2

TABLE 41
SAMPLE NUMBER MLO-05-421

## Rubber Swell Test (MIL-PRF-5606H, FTM 791C, method 36003) 168 Hours @ 70°C (158°F) using NBR-L(AMS 3217/2B) rubber specimens

	<b>Relative Density</b>	% Swelling
Specimen #1	1.192	35.2
Specimen #2	1.192	35.4
Specimen#3	1.192	35.2
Average:	1.192	35.3

TABLE 42
SAMPLE NUMBER MLO 06-0275

# RUBBER SWELL TEST MIL-PRF-5606H FTM 791, METHOD 3603

# $168 \ Hours \ @ \ 70^{o}C \ (158^{o}F)$ using AMS 3217/2B (NBR-L) rubber specimen

	<b>Relative Density</b>	% Swelling
Specimen #1	1.194	32.25
Specimen #2	1.193	32.16
Specimen #3	1.194	32.16
Average:	1.194	32.19

5. H	Iot	Surface	Ignition	<b>Determinations</b>
------	-----	---------	----------	-----------------------

**TABLE 43** 

# Hot Surface Ignition Temperature (ASTM D6688), °F

MLO-06-0280	1175
MLO-06-0281	1175
MLO-06-0282	1175
MLO-06-0283	1175

6. Autoignition Temperature Determinations		

#### **SAMPLE NUMBER MLO 03-10**

#### **Autoignition Temperature (ASTM E659)**

## **Hot Flame Autoignition Temperature (AIT)**

Minimum Ignition Temperature, °C(°F) 401.7(755)

Ignition Delay, sec. 8

Barometric Pressure, kPa(torr) 100.04(750.3)

#### **Cool-Flame Autoignition Temperature (CFT)**

Minimum Ignition Temperature, °C(°F) Not

Ignition Delay, sec. Observed

Barometric Pressure, kPa(torr)

# Reaction Threshold Temperature for Pre-flame reaction (RTT)

Minimum Reaction Temperature, °C(°F) 273.9(525)

#### **SAMPLE NUMBER MLO 03-25**

#### **Autoignition Temperature (ASTM E659)**

## **Hot Flame Autoignition Temperature (AIT)**

Minimum Ignition Temperature, °C(°F) 401.7(755)

Ignition Delay, sec. 12

Barometric Pressure, kPa(torr) 98.2(736.5)

#### **Cool-Flame Autoignition Temperature (CFT)**

Minimum Ignition Temperature, °C(°F) Not

Ignition Delay, sec. Observed

Barometric Pressure, kPa(torr)

# Reaction Threshold Temperature for Pre-flame reaction (RTT)

Minimum Reaction Temperature, °C(°F) 279.4(735)

7. Pressure Viscosity Testing Data	a Determinations

TABLE 46
SAMPLE NUMBER MLO-08-0221

# Pressure Viscosity, cP @ 125°F

Pressure, psi	Viscosity, cP
100	3.13
400	3.21
700	3.29
1000	3.37
1200	3.43
1500	3.51
1800	3.59
2000	3.65
3000	3.92
4000	4.20
5000	4.47

TABLE 47
SAMPLE NUMBER MLO-08-0221

# Pressure Viscosity, cP @ 100°F

Pressure, psi	Viscosity, cP
100	4.36
400	4.46
700	4.56
1000	4.66
1200	4.73
1500	4.83
1800	4.94
2000	5.00
3000	5.34
4000	5.68
5000	6.02

TABLE 48
SAMPLE NUMBER MLO-08-0221

# Pressure Viscosity, cP @ 77°F

Pressure, psi	Viscosity, cP
100	6.45
400	6.65
700	6.86
1000	7.06
1200	7.20
1500	7.40
1800	7.74
2000	7.74
3000	8.43
4000	9.11
5000	9.79

8. Vapor Pressure by Isoteniso	cope Determination	S	

TABLE 49
SAMPLE NUMBER MLO 04-1051

# Vapor Pressure by Isoteniscope (ASTM D2879), torr

Temperature, °F	Degassed with reflux	Degassed at RT
425	-	0.1
450	0.10	0.23
500	0.58	1.1
550	3.4	5.5
600	13.5	19.0
650	56	67
700	190	190
750	550	460

8.1 Vapor Pressure by Isoteniscope, Boiling Point and Heat of Vaporization Determinations

	MLO 07-329	MLO-07-330
Vapor Pressure by Isotenisco (ASTM D2879), torr	ppe	
Temperature, °F		
32	$(0.35 \times 10^{-2})$	$(0.15 \times 10^{-2})$
68	$(0.20 \times 10^{-1})$	$(0.88 \times 10^{-2})$
100	$(0.76 \times 10^{-1})$	$(0.34 \times 10^{-1})$
150	0.50	0.23
200	2.35	1.15
250	8.80	4.3
300	29	14.5
350	82	43
400	205	110
450	435	235
490	760	-
500	-	480
530	-	760
<b>Boiling Point (from</b>		
Vapor Pressure data), °F	490	530
Heat of Vaporization, kcal/mole		
From Antoine Equation	13.83	13.89
From Clausius Clapeyron		
Equation	13.81	14.14

	MLO 07-331	MLO-86-348
Vapor Pressure by Isotenis (ASTM D2879), torr	cope	
Temperature, °F		
32	$(0.56 \times 10^{-3})$	$(0.26 \times 10^{-4})$
68	$(0.35 \times 10^{-2})$	$(0.18 \times 10^{-3})$
100	$(0.14 \times 10^{-1})$	$(0.80 \times 10^{-3})$
150	0.10	$(0.66 \times 10^{-2})$
200	0.51	$(0.37 \times 10^{-1})$
250	2.05	0.16
300	7.2	0.60
350	21.5	1.95
400	56	5.4
450	125	12.5
500	260	27
550	540	59
580	760	-
600	-	110
650	-	250
Initial Decomposition Temperature, °F	-	620
Boiling Point (from Vapor Pressure data), °F	580	Note 1
Heat of Vaporization, kcal/mole		
From Antoine Equation	14.28	14.66
From Clausius Clapeyron Equation	14.50	15.58

Note 1: Sample decomposes at 620°F and 140 torr. The boiling point (760 torr can, therefore, not be determined at 1 atm.

9. Dielectric Strength Determinations

**TABLE 52** 

MLO 87-0222	MLO 04-060	MLO 06-0160

# Dielectric Strength (ASTM D877)

Breakdown #		kV, AC	
1	46.2	43.8	50.8
2	44.4	40.0	55.9
3	48.5	41.5	52.9
4	45.1	47.6	48.8
5	43.9	44.9	55.4
Average:	45.6	43.6	52.8
Standard Devia	<b>ntion:</b> 1.83	2.96	3.02

**TABLE 53** 

MLO 07-0262	MLO 07-0279	MLO 07-0280

# **Dielectric Strength (ASTM D877)**

Breakdown #		kV, AC	
1	70.5	57.6	58.8
2	37.5	60.1	50.2
3	61.8	48.2	56.2
4	36.4	57.2	54.0
5	48.1	51.4	55.1
6	45.4	58.9	
7	50.8	55.9	
8	40.9	57.4	
9	60.6	60.7	
10	42.3	44.4	
Average:	49.4	55.2	54.9
Standard Dev	viation: 11.44	5.41	3.16

 $<sup>\</sup>ast$  The average of 5 breakdowns did not meet paragraph 11.3.2 Acceptable Range Criteria.

**TABLE 54** 

MLO 06-0701

MLO 90-0715

MLO 06-0700

MLO 87-0222

		Dielectric Strength (ASTN	M D877)	
Breakdown #		Voltage, k	V, AC	
1	37.3	59.6	58.9	28.5
2	35.7	55.2	42.3	27.1
3	35.9	61.8	57.2	31.7
4	35.3	44.7	50.3	29.5
5	34.8	50.3	43.5	28.9
6		60.0	60.4	
7		62.9	34.5	
8		56.2	56.8	
9		60.3	56.5	
10		52.4	53.1	
Average:	35.8	56.3 (Note 1)	51.4 (Note 1)	29.1
Standard Deviation:	0.9	5.79	8.57	1.68

Note 1: The average of 5 breakdowns did not meet paragraph 11.3.2 Acceptable Range Criteria

<b>10.</b>	<b>Fretting</b>	Wear	<b>Test</b>	<b>Determinations</b>
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## **SAMPLE NUMBER MLO 04-70**

Fretting Wear Test	
(ASTM D4170), mass loss, mg	
Upper Race	16.5
Lower Race	25.8
Total	42.3
Mean	21.2

# **SAMPLE NUMBER MLO 04-6**

Fretting Wear Test	
(ASTM D4170), mass loss, mg	
Upper Race	9.2
Lower Race	11.4
Total	20.6
Mean	10.3

## **SAMPLE NUMBER MLO 03-278**

Fretting Wear Test	
(ASTM D4170), mass, mg	
Upper Race	10.1
Lower Race	2.5
Total	12.6
Mean	6.3

SAMPLE NUMBER	MLO 03-48	MLO 01-374
Fretting Wear Test		
(ASTM D4170), mass loss, mg		
Upper Race	6.5	8.9
Lower Race	3.6	11.7
Total	10.1	20.6
Mean	5.0	10.3

SAMPLE NUMBER	MLO 04-70	MLO 04-71
Fretting Wear Test		
(ASTM D4170), mass loss, mg		
Upper Race	3.1	10.2
Lower Race	3.7	10.3
Total	6.8	20.5
Mean	3.4	10.3

TABLE 60 Fretting Wear (ASTM D4170), mass loss, mg

	<b>Upper Race</b>	Lower Race	Total	Mean
PAO Type Greases				
MLO 04-643	2.7	1.8	4.5	2.2
MLO 04-644 Run 1 Run 2	9.3 16.7	12.3 14.9	21.6 31.6	10.8 15.8
MLO 04-645	12.6	17.2	29.8	14.9
MLO 04-646 Run 1 Run 2	4.3 10.5	5.2 5.1	9.5 15.6	4.8 7.8

11. Various Corrosion Testing Dat	11.	Various	<b>Corrosion</b>	<b>Testing</b>	Data
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# 11.1 Bearing Rust Test

**SAMPLE NUMBER MLO 01-127** MLO 01-232 Bearing Rust Test with 15% Synthetic Sea Water (ASTM D5969) 24 hrs. Pass Pass Heavy stain **SAMPLE NUMBER** MLO-01-127 MLO-01-232 **Bearing Corrosion Test** 10% Sea Water, 24 hrs. (Note 1) Pass (Note 2) (ASTM D5969) Pass

Note 1: New Bearing Holders

Note 2: Heavy Stain

**SAMPLE NUMBER** MLO-01-127 MLO-01-232 **Corrosion Prevention** (ASTM D1743) Pass **Pass** Bearing Rust Test with 100% Synthetic Sea Water (ASTM D5969) Fail Fail Bearing Rust Test with 50% Synthetic Sea Water (ASTM D5969) Fail Fail (Note 1) Bearing Rust Test with 25% Synthetic Sea Water

Fail (Note 2)

Fail (Note 1)

Note 1: (MLO-01-232 significantly worse than MLO-01-127)

Note 2: (2 out of 3 bearing races)

(ASTM D5969)

SAMPLE NUMBER MLO-01-127 MLO-01-232

Bearing Rust Test with 100% Synthetic Sea Water 24 hrs. in Large Jar (ASTM D5969)

Pass Pass

Bearing Rust test with 100% Synthetic Sea Water 24 hrs. in Small Jar (ASTM D5969)

Pass (Note 1) Fail (Note 2)

Note 1: 1 pass, 1 marginal, 1 fail.

Note 2: Severe stain all three rings.

SAMPLE NUMBER	MLO-94-23	MLO-03-48

Bearing Rust Test with 100% Synthetic Sea Water 24 hrs. in Large Jar (ASTM D5969)

Pass Pass

Bearing Rust test with 100% Synthetic Sea Water 24 hrs. in Small Jar (ASTM D5969)

Pass/Fail (Note 1) Fail

Note 1: 1 pass, 1 marginal, 1 fail.

#### Part I

ASTM D1743 and ASTM D5969 are basically identical tests, except:

#### **ASTM D1743**

Determining Corrosion Preventive Properties of Lubricating Grease (Bearing Corrosion) is run with deionized water for 48 hours.

#### **ASTM D5969**

Corrosion Preventive Properties of Lubricating Grease in Presence of Dilute Synthetic Sea Water Environments (Bearing Corrosion) is run with synthetic sea water for 24 hours.

MLO-01-127 and MLO-01-232 both passed ASTM D1743.

Tested under ASTM D5969:

Concentration of Sea Water:	MLO-01-127	MLO-01-232
100%	Fail	Fail
70%	Fail	Fail
50%	Fail	Fail (Note 1)
25%	Fail (Note 2)	Fail (Note 1)

Note 1: Significantly worse than MLO-01-127.

Note 2: Two out of three bearing rings failed.

#### Part II

MLO-94-23 was run only as ASTM D4693 (Low Temperature Torque of Grease-Lubricated Wheel Bearings). Results are reported in N·m.

ASTM D1478 (Low Temperature Torque of Ball Bearing Grease) results are reported in gcm).

#### Investigation of Jar Sizes Used in ASTM D1743 & ASTM D5969

- 1. It was found that neither test method includes the dimensions for plastic jars used nor the dimensions of the individual parts of that make up the Bearing Holder Assembly.
- 2. Figure 1, of either method (see attached), is also ambiguous in regards to what is actually available from a particular supplier<sup>1.</sup> While Figure 1 shows a gap, of unknown size, between the Bearing Holder (8) and the side wall of the Plastic Jar (9), this has created confusion as to what is or is not the intended size of jar.

(Assemblies from the supplier provide an extremely small gap between the holder and side wall).

- 3. Ultimately, tests performed in different sized jars have provided different results. Initially, tests were performed in 1000 mL (32 oz.) jars resulted in a Pass rating. Subsequent tests performed in 500 mL (16 oz.) jars resulted in a Fail rating.
  - a. In accordance with either method, the specified amount of water (100±5 mL) injected into the injection/extraction hole will completely immerse a bearing holder in the 500 mL jar but not in the 1000 mL jar.
  - b. While 100 mL is sufficient to provide total immersion in the 500 mL jar, 250 mL is required to provide equivalent coverage in a 1000 mL jar.

In retrospect, after extraction, would a remaining amount of 75±5 mL have been required to provide the appropriate bottom water (i.e. approximately 30% of the injected volume), or a remainder of 60±5 mL to remain proportional to a 500 mL jar volume?

- 4. The 1000 mL plastic jars used are approximately 5.5" tall and 4.4 " in diameter at the opening with a very slight taper at the bottom.
- 5. The 500 m jars, from the same manufacturer, may be either 4.0 or 3.75" tall and 3.20 or 3.25" respectively in diameter at the opening with a *slight* taper at the bottom.

Conclusion: The omission of dimensions has led to confusion for any independent laboratory wishing to fabricate the test apparatus on site as well as a difference in test results.

<sup>&</sup>lt;sup>1</sup> Koehler Instrument Company, 1595 Sycamore Avenue, Bohemia, NY 11716

# **SAMPLE NUMBER MLO 06-232**

Corrosion Prevention (ASTM D5969), @ 5% Synthetic Sea Water

Fail

# 11.2 Copper Strip Corrosion

MLO 06-0457 MLO 05-0967

Copper Strip Corrosion (ASTM D4048)

24 hours @ 212°F 4A, Corrosion 4A, Corrosion

#### SAMPLE NUMBER MLO 05-266 (MIL-PRF-6086)

Copper Strip Corrosion per MIL-PRF-6086 (ASTM D130) 3 hrs. @ 100°C

Moderate Tarnish, 2C

# 12. Miscellaneous Studies

SAMPLE NUMBER	MLO 94-23	MLO 02-20
Bomb Oxidation Test		
(ASTM D942) 500 hrs.		
pressure drop, psi	22	18

SAMPLE NUMBER	MLO 03-232	MLO 03-233
Evaporation Loss (ASTM D972), 100°C, 22 hrs, % Run 1 Run 2	18.9 19.2	20.4 20.8
Acid No. (ASTM D664), mgKOH/gram	1.58	1.67

#### **SAMPLE NUMBER MLO 03-48**

Dirt Count (FTM STD. 791, 3005.4), particles/cm <sup>3</sup>	
25 microns & larger	58
75 microns & larger	0
125 microns & larger	0

#### 4-Ball Wear Test (ASTM D2266) 40 Kg, 1200 RPM, 1 hr. @ 167°F Average wear scar diameter, mm

MLO-07-0037	1.19
MLO-07-0038	1.66
MLO-07-0039	1.39

4-Ball Wear Test (ASTM D2266) (Modified), 10Kg for 1 minute, 20 Kg for 1 minute, 30 KG for 1 minute finish test at 40 Kg for 57 minutes, 1200 RPM, @ 167°F Average wear scar diameter, mm

MLO-07-0037	1.09
MLO-07-0038	0.99
MLO-07-0039	1.39

SAMPLE NUMBER	MLO 03-26	MLO 03-27
4-Ball Wear Test		
(MIL-PRF 5606)		
40 KG, 1200 RPM,		
75°C, 1 hr.		
Average Wear Scar Diameter, mm	1.01	0.86

#### **SAMPLE NUMBER MLO 01-127**

Water Resistance (ASTM D1264) @ 41°C % washout	2.75
Low Temperature Torque (ASTM D1478) @ -40°C(-40°F) Starting Torque, g-cm Running Torque (10 min), g-cm Running Torque (60 min), g-cm	>14,750 >14,750 8,998
Corrosion Prevention (ASTM D1743)	Pass (No Rust)
Bearing Rust Test, With Synthetic Sea Water (ASTM D5969)	Pass (No Rust)
Fretting Wear (ASTM D4170) mass loss, mg Upper Race Lower Race Total	0.9 0.4 1.3
Bomb Oxidation Stability (ASTM D942) 500 hours pressure drop, psi	
Dirt Count FTM 791.3005.4, particles/cm <sup>3</sup> 25 microns & larger 75 microns & larger 125 microns & larger	5 144 24 0

# **SAMPLE NUMBER MLO 02-326**

Water Resistance (ASTM D1264) @ 41°C % washout	2.0
Low Temperature Torque (ASTM D1478) @ -40°C(-40°F) Starting Torque, g-cm Running Torque (10 min), g-cm Running Torque (60 min), g-cm	1416 620 295
Corrosion Prevention (ASTM D1743)	Pass (No Rust)
Bearing Rust Test, With Synthetic Sea Water (ASTM D5969)	Fail
Fretting Wear (ASTM D4170) mass loss, mg Upper Race Lower Race Total	3.9 5.9 9.8
Bomb Oxidation Stability (ASTM D942) 500 hours pressure drop, psi Run 1 Run 2	25 19
Dirt Count FTM 791.3005.4, particles/cm <sup>3</sup> 25 microns & larger 75 microns & larger 125 microns & larger	100 25 0

# **TABLE 72 CONTINUED**

#### **SAMPLE NUMBER MLO-02-326**

#### **LOAD WEAR INDEX (ASTM D2596)**

Applied Load, Kgf	Scar Diameter, mm	Corrected Load, Kgf
40	0.32*	
50	0.38	42.318
63	1.72	12.723
80	1.99	15.122
100	2.03	19.961
126	2.29	24.081
200	Weld	
Total of Corrected Loads	•	114.205
Total of Compensation L	ine Corrected Loads	118.0
Total A		232.205

Total A 232.205  
LOAD WEAR INDEX, kgf =---- = 23.2  

$$10$$
  $10$ 

# **SAMPLE NUMBER MLO 03-21**

Water Resistance	
(ASTM D1264) @ 41°C	
% washout	0.5
Low Temperature Torque	
(ASTM D1478) @ -40°C(-40°F)	
Starting Torque, g-cm	5369
Running Torque (10 min), g-cm	2714
Running Torque (60 min), g-cm	1062
Bearing Rust Test with Distilled Water	
(ASTM D1743)	Pass (No Rust)
Bearing Rust Test with Synthetic Sea Water(100%)	
(ASTM D5969)	Pass (No Rust)
Fretting Wear (ASTM D4170),	
Mass loss, mg	
Upper Race	1.5
Lower Race	1.4
Total	2.9
Bomb Oxidation Stability	
(ASTM D942), 500 hrs.	
pressure drop, psi	2
Dirt Count	
FTM 791.3005.4, particles/cm <sup>3</sup>	
25 microns & larger	135
75 microns & larger	54
125 microns & larger	27
Worked Penetration (ASTM D217)	
60 strokes	293

# **TABLE 73 CONTINUED**

#### **SAMPLE NUMBER MLO-03-21**

#### **LOAD WEAR INDEX (ASTM D2596)**

Applied Load, Kgf	Scar Diameter, mm	Corrected Load, Kgf
200	0.46*	
126	2.23	24.729
160	2.36	32.131
200	Weld	
Total of Corrected Loads		56.860
Total of Compensation L	ine Corrected Loads	364.5
Total A		421.360

SAMPLE NUMBER	MLO 03-155	MLO 03-156
Water Resistance (ASTM D1264) @ 41°C % washout	1.0	0.75
Low Temperature Torque (ASTM D1478) @ -40°C(-40°F) Starting Torque, g-cm	5162	944
Running Torque (60 min), g-cm	560	354
Corrosion Prevention (ASTM D1743)	Pass	Pass
Bearing Rust Test with Synthetic Sea Water (ASTM D5969) 24 hrs.	Fail	Fail
Fretting Wear (ASTM D4170) mass loss, mg		Run 1 Run 2
Upper Race Lower Race Total	5.2 5.7 10.9	12.2     11.7       10.1     14.2       22.3     25.9
Bomb Oxidation Stability (ASTM D942) 500 hours		
pressure drop, psi	6	4
Dirt Count FTM 791.3005.4, particles/cm <sup>3</sup>		
25 microns & larger	87	95
75 microns & larger	0	0
125 microns & larger	0	0

# **TABLE 74 CONTINUED**

#### **SAMPLE NUMBER MLO-03-155**

#### **LOAD WEAR INDEX (ASTM D2596)**

Applied Load, Kgf	Scar Diameter, mm	Corrected Load, Kgf
40	0.31*	
50	0.38	42.318
63	0.66	33.158
80	1.50	20.062
100	1.67	24.264
126	1.81	30.467
200	2.70	32.827
250	Weld	37.817
Total of Corrected Loads		220.913
Total of Compensation Li	ine Corrected Loads	86.4
Total A		307.313

Total A 307.313  
LOAD WEAR INDEX, kgf = 
$$\frac{12}{10}$$
 10

# **TABLE 74 CONTINUED**

#### **SAMPLE NUMBER MLO-03-156**

#### **LOAD WEAR INDEX (ASTM D2596)**

Applied Load, Kgf	Scar Diameter, mm	Corrected Load, Kgf
50	0.49	32.818
63	0.43	50.894
80	0.80	37.616
100	1.32	30.698
126	1.44	38.295
160	1.71	44.345
201	1.87	54.603
250	2.06	66.742
315	2.18	85.830
400	2.72	94.593
500	Weld	
Total of Corrected Loads		536.434
Total of Compensation L	ine Corrected Loads	0.0
Total A		536.434

# TABLE 75 SAMPLE NUMBER MLO 05-0967

Low	Temperature Torque, (ASTM D1478)			
	Torque, g.cm	@ -40°C		@ -54°C
	Starting Torque, g-cm	3,392		13,718
	Running Torque (1 min), g-cm	2,802		10,414
	Running Torque (10 min), g-cm	2,360		7,316
	Running Torque (60 min), g-cm	708		2,950
	Count, particles/cm <sup>3</sup> CM Std 791C, 3005.3)			
	25 microns and larger		26	
	75 microns and larger		0	
	125 microns and larger		0	
	ng Wear (ASTM D4170), MIL=PRF-32014			
	Upper Race		12.5	
	Lower Race		12.9	

#### **SAMPLE NUMBER MLO 05-967**

Water Resistance @ 41°C (ASTM D1264), % Washout	0.50	
Corrosion Prevention (ASTM D1743)		Pass (No Rust)
Corrosion Prevention (ASTM D5969) 5% Synthetic Sea Water		Fail
Low Temperature Torque, (ASTM D1478) Torque, g.cm	@ -40°C	@ -54°C
Starting Torque, g-cm Running Torque (1 min), g-cm Running Torque (10 min), g-cm Running Torque (60 min), g-cm	3,982 2,950 2,036 590	10,325 8,702 7,080 2,950
Dirt Count, particles/cm <sup>3</sup> (FTM Std 791C, 3005.3)		
25 microns and larger 75 microns and larger 125 microns and larger		78 0 0
Fretting Wear (ASTM D4170) Upper Race Lower Race		6.6 20.4
Bomb Oxidation Stability (ASTM D942) @ 210°F, pressure drop, psi		12
100 hrs. 220 hrs. 320 hrs. 420 hrs. 500 hrs.		12 20 22 23 28.5
# 4 4 === 4.		

#### **SAMPLE NUMBER MLO-05-967**

#### **LOAD WEAR INDEX (ASTM D2596)**

Applied Load, Kgf	Scar Diameter, mm	Corrected Load, Kgf
80	0.42*	
100	1.46	27,754
126	1.56	35.350
160	1.84	41.212
200	1.96	52.095
250	1.97	69.791
315	2.48	75.447
400	3.20	80.404
500	Weld	
Total of Corrected Loads		382.053
Total of Compensation L	ane Corrected Loads	172.4
Total A		554.453

#### **SAMPLE NUMBER MLO 06-232**

Water Resistance @ 41°C (ASTM D1264), % Washout	0.77
Dirt Count, particles/cm <sup>3</sup> (FTM Std 791C, 3005.3)	
25 microns and larger 75 microns and larger 125 microns and larger	182 52 0
Bomb Oxidation Stability (ASTM D942) @ 210°F, pressure drop, psi	
100 hrs.	1
268 hrs.	1
412 hrs.	1
500 hrs.	3
Low Temperature Torque, (ASTM D1478) Torque, g.cm	
1 / 2	@ -54°C
Starting Torque, g-cm Running Torque (1 min), g-cm Running Torque (10 min), g-cm Running Torque (60 min), g-cm	20,650 12,538 4,572 3,658
Corrosion Prevention (ASTM D1743)	Pass (No Rust)
Corrosion Prevention (ASTM D5969) 25% Synthetic Sea Water	Fail

# **TABLE 77 CONTINUED**

#### **SAMPLE NUMBER MLO-06-232**

#### **LOAD WEAR INDEX (ASTM D2596)**

Applied Load, Kgf	Scar Diameter, mm	Corrected Load, Kgf
63	0.36*	
80	1.35	22.291
100	1.29	31.412
126	1.53	36.043
160	1.82	41.665
200	1.93	52.905
250	2.15	63.948
315	2.36	79.284
400	2.97	86.631
500	Weld	
Total of Corrected Loads		414.179
Total of Compensation L	ine Corrected Loads	100.8
Total A		514.979

Total A 514.979  
LOAD WEAR INDEX, kgf =---- = 51.5  

$$10$$
 10

#### **SAMPLE NUMBER MLO 06-233**

Water Resistance @ 41°C	
(ASTM D1264), % Washout	1.24
Dirt Count, particles/cm <sup>3</sup> (FTM Std 791C, 3005.3)	
25 microns and larger	494
75 microns and larger	130
125 microns and larger	0
Bomb Oxidation Stability (ASTM D942) @ 210°F, pressure drop, psi	
100 hrs.	1
268 hrs.	1.5
412 hrs.	1.5
500 hrs.	1.5
Low Temperature Torque, (ASTM D1478) Torque, g.cm	
1 / 5	@ -54°C
Starting Torque, g-cm	10.325
Running Torque (1 min), g-cm	6.048
Running Torque (10 min), g-cm	2.212
Running Torque (60 min), g-cm	2.212
Corrosion Prevention (ASTM D1743)	Pass (No Rust)
Corrosion Prevention (ASTM D5969)	Dage (No Deve)
25% Synthetic Sea Water	Pass (No Rust)

# **TABLE 78 CONTINUED**

#### **SAMPLE NUMBER MLO-06-233**

#### **LOAD WEAR INDEX (ASTM D2596)**

Applied Load, Kgf	Scar Diameter, mm	Corrected Load, Kgf
63	0.37*	
80	1.26	23.883
100	1.44	28.140
126	1.60	34.466
160	1.71	44.345
200	1.83	55.796
250	2.03	67.728
315	2.14	87.434
400	2.55	100.899
500	3.39	102.198
620	Weld	
Total of Corrected Loads	S	544.889
Total of Compensation L	ine Corrected Loads	56.1
Total A		600.989

#### **SAMPLE NUMBER MLO 06-0491**

Water Washout @ 41°C	
(ASTM D1264), %	3.58
Bomb Oxidation Stability (ASTM D942)	
@ 210°F, pressure drop, psi	
100 hrs.	0
264 hrs.	0
384 hrs.	2.5
500 hrs.	4
Low Temperature Torque, (ASTM D1478)	
Torque, g.cm	
	@ -54°C
Starting Torque, g-cm	19,028
Running Torque (1 min), g-cm	12,832
Running Torque (10 min), g-cm	5,605
Running Torque (60 min), g-cm	3.,982
Dirt Count, particles/cm <sup>3</sup> (FTM Std 791C, 3005.3)	
25 microns and larger	93
75 microns and larger	0
125 microns and larger	0

# **TABLE 79 CONTINUED**

#### **SAMPLE NUMBER MLO-06-0491**

# **LOAD WEAR INDEX (ASTM D2596)**

Applied Load, Kgf	Scar Diameter, mm	Corrected Load, Kgf
100	0.44*	25.890
126	2.13	33.259
160	2.28	
200	Weld	
Total of Corrected Loads		59.149
Total of Compensation Line Corrected Loads		364.5
Total A		423.649

#### **SAMPLE NUMBER MLO 03-48**

@ 41°C, % washout	3.0
Bearing Corrosion Test (ASTM D5	969)
100% Synthetic Sea Water, 24 hour	S.
Bearing Cup #1	Fail. (1-1 mm long spot)
Bearing Cup #2	Fail. (1-2 mm long spot)
Bearing Cup #3	Fail. (3-2 mm long spots)
Bearing Corrosion Test (ASTM D1	743)
Deionized water, 48 hours.	
Bearing Cup #1	Pass.
Bearing Cup #2	Pass.
Bearing Cup #3	Pass.
Fretting Wear (ASTM D4170)	
mass loss, mg	

16.4

25.8

42.2

21.1

Bomb Oxidation Stability (ASTM D942) 500 hrs.

Upper Race

Lower Race

Total

Mean

pressure drop, psi 4

Water Resistance (ASTM D1264)

# **TABLE 80 CONTINUED**

#### **SAMPLE NUMBER MLO-03-48**

# **LOAD WEAR INDEX (ASTM D2596)**

Applied Load, Kgf	Scar Diameter, mm	Corrected Load, Kgf
63	0.38*	
80	0.43	69.984
100	1.94	20.887
126	2.17	25.413
Total of Corrected Loads		116.284
Total of Compensation Line Corrected Loads		218.8
Total A		335.084

Total A 335.084  
LOAD WEAR INDEX, kgf = 
$$\frac{16}{10}$$
 10

	MLO 07-0363 (SPECTRASYN 2C)	MLO 07-0364 (SPECTRASYN 4)	
Density, grams/cm <sup>3</sup>			
@ 100°C	0.7441	0.7651	
@ 200°C	0.6780	0.7035	
@ 300°C	0.6107	0.6341	
Latent Heat of Vaporization, kcal/r	nol		
Clausius-Clapeyron Equation	on 13.76	14.98	
Antoine Equation	13.71	15.03	
Vapor Pressure by Isoteniscope (ASTM D2879), torr			
Temperature, °F			
32 68 100 150 200 250 300 350 400 450 500 550 600 640 Initial Decomposition Temperature, °F	(0.64x10 <sup>-3</sup> ) (0.37x10 <sup>-2</sup> ) (0.14x10 <sup>-1</sup> ) (0.86x10 <sup>-1</sup> ) 0.39 1.45 4.7 13 33 70 140 285 490 760	(0.42x10 <sup>-4</sup> ) (0.21x10 <sup>-3</sup> ) (0.21x10 <sup>-2</sup> ) (0.13x10 <sup>-1</sup> ) (0.65x10 <sup>-1</sup> ) 0.26 0.90 2.8 7.0 16 38	
(By extrapolation)			

	MLO 07-0363 (SPECTRASYN 2C)	MLO 07-0364 (SPECTRASYN 4)
Thermal Conductivity, cal/sec(cm) <sup>2</sup> (°C/cm)		
@ 100°C	32.3x10 <sup>-5</sup>	33.5x10 <sup>-5</sup>
@ 200°C	28.6x10 <sup>-5</sup>	31.3x10 <sup>-5</sup>
@ 250°C	27.9x10 <sup>-5</sup>	24.7x10 <sup>-5</sup>
Specific Heat (ASTM D2766)		
@ 100°C	0.554	0.615
@ 200°C	0.644	0.677
@ 300°C	0.735	0.740

**TABLE 82** 

	MLO 07-0648 (DC200 Silicone)	MLO 08-0013 (Silahydrocarbon)
Density, grams/cm <sup>3</sup> (ASTM D2766)		
@ 100°C	0.8016	0.7241
@ 200°C	0.7355	0.6418
@ 250°C	0.7009	Note 1
@ 300°C	0.6559	Note 1

Note 1: Density cannot be determined because sample is too volatile.

Thermal Conductivity, cal/sec(cm)<sup>2</sup>(°C/cm) (ASTM D2717

$(31.0x10^{-5})$	$(29.0 \times 10^{-5})$
$(27.9 \times 10^{-5})$	$(28.6 \times 10^{-5})$
$(26.9 \times 10^{-5})$	$(27.3 \times 10^{-5})$
	$(27.9 \times 10^{-5})$

<sup>\*</sup> Samples were too volatile to permit determination of thermal conductivity @ 300°C

Vapor Pressure by Isoteniscope (ASTM D2879), torr

Temperature, °F

32	$(0.27 \times 10^{-2})$	$(0.27 \times 10^{-1})$
68	$(0.11 \times 10^{-1})$	0.12
100	$(0.3x10^{-1})$	0.38
150	0.13	1.9
200	0.76	7.4
250	1.2	23
300	3.1	64
350	7.0	160
400	14.5	350
450	26	680
460	-	760
500	45	
550	77	

Initial Decomposition Temperature, °F 585

(By extrapolation)

# **TABLE 82 CONTINUED**

	MLO 07-0648 (DC200 Silicone)	MLO 08-0013 (Silahydrocarbon)
Specific Heat (ASTM D2766)		
@ 100°C	0.523	0.590
@ 200°C	0.651	0.626
@ 250°C	0.685	0.644
@ 300°C	0.720	0.662

**TABLE 83** 

	MLO 07-0451 (Silahydrocarbon)	MLO 07-0262 (alkylbenzene)
Density, grams/cm <sup>3</sup>		
@ 100°C	0.7636	0.7999
@ 200°C	0.7001	0.7372
@ 300°C	0.6275	0.6689
Latent Heat of Vaporization, kcal/m	ol	
Clausius-Clapeyron Equation	n 15.46	12.68
Antoine Equation	14.34	12.09
Vapor Pressure by Isoteniscope (AS Temperature, °F	TM D2879), torr	
32 68 100 150 200 250 300 350 400 450 500 550 600 Initial Decomposition Temperature, °F	(0.25x10 <sup>-4</sup> ) (0.17x10 <sup>-3</sup> ) (0.76x10 <sup>-3</sup> ) (0.64x10 <sup>-2</sup> ) (0.35x10 <sup>-1</sup> ) 0.15 0.56 1.8 5.0 11.5 24.5 53 96	(0.03x10 <sup>-3</sup> ) (0.15x10 <sup>-2</sup> ) (0.46x10 <sup>-2</sup> ) (0.26x10 <sup>-1</sup> ) 0.11 0.35 1.05 2.8 6.1 12 23 43
(By extrapolation)		

# **TABLE 83 CONTINUED**

	MLO 07-0451 (Silahydrocarbon)	MLO 07-0262 (alkylbenzene)
Thermal Conductivity, cal/sec(cm) <sup>2</sup> (°C/cm)		
@ 100°C	$35.4 \times 10^{-5}$	34.0x10 <sup>-5</sup>
@ 200°C	26.6x10 <sup>-5</sup>	$30.8 \times 10^{-5}$
@ 250°C	25.8x10 <sup>-5</sup>	24.8x10 <sup>-5</sup>
Specific Heat (ASTM D2766)		
@ 100°C	0.609	0.582
@ 200°C	0.611	0.625
@ 300°C	0.613	0.668

#### MLO-90-0655 (on 2)

#### Fluorinert Electron Liquid FC-72

Specific Heat (ASTM D2766)

@ 100°C The sample is too volatile to permit

@ 200°C specific heat determination at 100°C or 200°C

Density (ASTM D1475), grams/cm<sup>3</sup>

@ 30°C 1.6671

@ 100°C The sample is too volatile to permit density

@ 200°C determination at 100°C or 200°C.

Thermal Conductivity (ASTM D2717), cal/sec(cm)<sup>2</sup>(°C/cm)

@ 30°C
 @ 100°C
 The sample is too volatile to permit thermal conductivity determination at 30°C, 100°C

@ 200°C or 200°C.

Vapor Pressure by Isoteniscope (ASTM D2879), torr

@	0°C	90
	20°C	210
	38.8°C	395
	61.1℃	760

Heat of Vaporization @ 30°C, kcal/mol 6.26

#### Comment:

The physical properties of the sample at 100°C and 200°C could not be determined because the indicated test temperatures are above the boiling point of the sample.

# Thermal Conductivity @ 100°C, cal/sec(cm)<sup>2</sup>(°C/cm)

MLO-07-0329	$18.9 \times 10^{-5}$
MLO-07-0330	18.9x10 <sup>-5</sup>
MLO-07-0331	$18.5 \times 10^{-5}$
Specific Heat (ASTM D2766) @ 100°C	
MLO-07-0329	0.274
MLO-07-0330	0.277
MLO-07-0331	0.280

#### 13. References

#### 13.1 AFWL-TR-92-4020

A.A. Krawetz, Technical Report AFWL-TR-923-4020, Volume II, Thermochemical and Physical Behavior of Lubricants and Hydraulic Fluids, Air Force Systems Command (ASD), Wright Laboratory (ML/MLBT), Wright-Patterson Air Force Base, Ohio, May, 1992.

#### 13.2 WL-TR-96-4090

A.A. Krawetz, Technical Report WL-TR-96-4090, Thermochemical and Physical Properties of Fluids, Lubricants and Related Materials, Volume 3, Improved Corrosion Rate Evaluation Procedure; Chemical and Physical Properties of Lubricants and Hydraulics Fluids, May, 1996.

#### 13.3 AFRL-ML-WP-TR-2002-4185

A.A. Krawetz, Technical Report AFRL-ML-WP-TR-2002-4185, Thermochemical and Physical Properties of Fluids, Lubricants and Related Materials, Volume 1, Improved Corrosion Rate Evaluation Procedure, August 2002.